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(54) Name of the invention:

Adhesive Tape and Its Manufacturing Method

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(54) [Name of the Invention]

Adhesive Tape and Its Manufacturing Method

(57) [Abstract]

[Objective]

The objective of the present invention is to obtain an adhesive tape that is comprised of a 0.2 mm or more, thick, foam like adhesive agent layer, which is compressible and also has restoring properties, and of a skin layer.

[Structure]

The structure of the adhesive tape is formed from a foam type adhesive agent layer, which contains fine elastic particles, and from a skin layer, which contains a reinforced material impregnated with the adhesive agent. The manufacturing method for the preparation of the adhesive tape is comprised of the technological process of coating an ultraviolet light curable type adhesive agent composition material on the surface of the placed release type substrate material, the technological process of layer laminating sequentially a reinforced substrate material and a transparent release film on the surface of the ultraviolet light curable type adhesive agent coated layer, and the technological process of irradiating an ultraviolet light from above the transparent release film side.

[Scope of the Claims]

[Claim 1]

Adhesive tape characterized by the fact that it is formed from an adhesive agent layer, which contains fine elastic particles, and from a reinforced material impregnated with an adhesive agent.

[Claim 2]

Adhesive tape according to the above Claim 1 characterized by the fact that the fine, elastic properties possessing particles are hollow fine particles, which use an elastomer material as their wall material.

[Claim 3]

A manufacturing method for the preparation of the adhesive tape, which is comprised of the technological process of coating an ultraviolet light curable type adhesive agent composition material on the surface of the placed release type substrate material, the technological process of layer laminating sequentially a reinforced substrate material and a transparent release film on the surface of the ultraviolet light curable type adhesive agent coated layer, and the technological process of irradiating an ultraviolet light from above the transparent release film side.

[Claim 4]

Manufacturing method for the preparation of the adhesive tape according to the above Claim 3, characterized by the fact that the reinforced material does not allow the passing of the fine, elastic particles.

[Detailed Explanation of the Invention]

[0001]

[Technical Field of the Invention]

This invention is an invention about a pressure sensitive adhesive tape using an ultraviolet light curable type adhesive agent composition material,, and it is an invention about its manufacturing method.

[0002]

[Prior Art]

In the past, the adhesive tapes have been single sided adhesive tapes where on one surface of plastic film, paper, fabric or metal foil, an adhesive agent layer has been provided, or double sided tapes, where an adhesive agent layer has been provided on a release type substrate material. Within the double sided adhesive tape category, there is the tapes that do not use reinforced substrate materials and the tapes that use the reinforced substrate materials.

[0003]

The former category is a material obtained as an adhesive agent has been coated on the surface of a release type substrate material, and dried, and it is called non-support or transfer type adhesive tape. Also, in the description reported according to the Japanese Patent Application Laid Open Number Showa 56-159269, etc., the manufacturing method for the preparation of a double sided adhesive tape has been disclosed whereby an adhesive agent where a foaming agent has been admixed, is coated on a release type

substrate material, and after that it is heated and foamed. In the description reported according to the Japanese Patent Application Laid Open Number Showa 63-72532, also, a double sided adhesive tape has been introduced where hollow thermoplastic spheres have been mixed in the adhesive agent and a foam like structure was obtained.

[0004]

The latter category is a material that is formed as an adhesive agent layer is formed on both sides of a reinforced material substrate, like for example, paper, nonwoven fabric, polyester film or urethane foam, chloroprene foam, ethylene/vinyl chloride copolymer material foam, polyethylene foam, etc., film or sheet material. Also, as it can be seen from the description reported according to the Japanese Patent Application Laid Open Number Hei-Sei 6-189316, applications relative to foam type adhesive tape where adhesive agent layer containing fine, elastic particles, has been reinforced by a number of substrate materials, have also been filed.

[0005]

Regarding the preparation of the double sided adhesive tape, there is the method where adhesive agent layers are formed directly on both surfaces of the reinforcing material substrate, and dried, and the method where the material obtained as an adhesive agent has been formed on a release substrate material, is layer laminated on both sides of a reinforced substrate material. In the double sided adhesive tape, it is possible to use a solvent agent type, an emulsion type, a hot melt type or an ultraviolet curable type adhesive agent.

[0006]

Regarding the solvent agent type and the emulsion type adhesive agent, it is a material obtained as at least one type of meth (alkyl) acrylate where the number of the carbon atoms is in the range of 4 ~ 12 (ester of an alcohol, where the number of the carbon atoms is in the range of 4 ~ 12, and a (meth)acrylic acid) and a compound copolymerizable with that, are dispersed in a solvent medium or water and polymerized. The hot melt type adhesive agent is a material that is prepared as to olefin type thermoplastic resin adhesion imparting resins which have different softening points, are added and the melting temperature is adjusted. And the ultraviolet light curable type adhesive agent is a material obtained as a photoinitiator is added to a composition material that has as its main component, an alkyl (meth)acrylate that is the same as the above described type.

[0007]

For the methods for the preparation of the adhesive tapes, there are the method whereby a solvent agent type or an emulsion type adhesive agent is coated on a supporting material or on a release type substrate material, and after that the solvent agent or the water is removed, the method whereby a hot melt adhesive agent is heated and melted and by

using an extruder it is extruded on the surface of a supporting material or a release type substrate material, etc.

[0008]

In the case of the ultraviolet curable type adhesive agent, different methods have been disclosed like the method whereby an ultraviolet light is irradiated towards a composition material that is formed from an alkyl (meth)acrylate and a compound that is copolymerizable with that (Belgium patent Number 675,420,5/1966), the method whereby an ultraviolet light with a wavelength in the range of 300 ~ 400 nm (it is also a good option if it contains 10 % ultraviolet light with a wavelength of 300 nm or less) is irradiated towards an adhesive agent composition material that has an acrylic type monomer as its main component, at an intensity of 7 mW/cm² or less (USA Patent Number 4,181,752) etc., methods.

[0009]

[Problems Solved by the Present Invention]

Usually, the adhesive agent has adhesive properties and it shows flow properties and because of that it looks soft, however, at the time of the adhesion (gluing) it is difficult to be compressed by the pressure and because of that when an adhesive agent layer is placed between two materials subjected to the adhesion that do not have an even flatness, it is difficult to close the gap even by compressing applying a high pressure force. Especially, even if by applying a high pressure force it is possible to close the gap, the material subjected to the adhesion is deformed, and also, after some time after the pressure adhesion, the deformation of the material subjected to the adhesion is restored and the gap between the adhered two materials subjected to the adhesion is generated and this becomes a source of floating or separation. Also, there is a generation of bleed out of the adhesive agent and that is why it is not preferred.

[0010]

As the adhesive tape that can be compressed, foamed film or sheet reinforced material, for example, urethane foam, chloroprene foam, ethylene/vinyl acetate copolymer foam, polyethylene foam, etc., utilizing double side adhesive foam tape is used to adhere the above described material subject to the adhesion, and effective results have been observed, however, because of the fact that the adhesive agent layer is glued on both sides of the foam substrate material, the adhesive force between the substrate material and the adhesive agent is insufficient and there is separation and there have been problems related to the durability properties of the foam itself.

[0011]

Also, in the case of the double sided tape using a foaming agent there are the drawback points that it has been stated that due to gas absorption the thickness cannot be

maintained constant and the regeneration properties are also lost. On the other hand, in the case of the foam type pressure sensitive adhesive agent covered sheet material, disclosed according to the practical example in the description reported in the Japanese Patent Application Laid Open Number Showa 63-72532, where hollow thermoplastic spheres have been admixed inside the adhesive sheet, the regeneration properties after the compression and the adhesive properties relative to rough surfaces are excellent, however, because of the fact that spheres are present at the front surface of the sheet, the smoothness properties are lost, and this is apparent at the surface of the material that is subjected to the adhesion. Especially, at the time when the adhesive agent layer, containing the spheres, is punched to the predetermined size, and by using a blade the adhesive agent is glued, because of the fact that the material subject to the adhesion is not accommodating, it is easy to be extended.

[0012]

The present invention is an invention that has as a goal to solve the above described problems according to the previous technology, and it has the goal to obtain an adhesive tape that has the described here below properties.

[0013]

- (1) An adhesive tape that has a foam type surface with a thickness of at least 0.2 mm or higher, and a skin surface from a reinforced substrate material.
- (2) Pressure adhesive tape which is compressible and which regenerates (restores).
- (3) Adhesive tape, which adheres intimately and strongly two materials subjected to the adhesion, which have a gap between them that is not constant.
- (4) Adhesive tape, which has good pull-out processing properties and adhesion operation handling properties.
- (5) Adhesive tape, which has high adhesion strength and a smooth adhesion surface.
- (6) Adhesive tape, which is obtained by using an ultraviolet light curable type adhesive agent, and which is obtained by a manufacturing process, that is inexpensive and that does not pollute the environment.

[0014]

[Measures in Order to Solve the Problems]

The authors of the present invention have conducted rigorous research in order to achieve the above described goal, and as a result from that as it is shown according to the presented in Figure 1, they have achieved the completion of this goal by a method where an adhesive tape, formed from the adhesive agent layer 8, which contains the micro-balloons (hollow fine particles) 4 and the polyolefin powder 5, and the adhesive agent layer 6, which is placed after the reinforced substrate material 3 is passed, is cured by ultraviolet light radiation.

[0015]

Regarding the adhesive tape according to the present invention, the micro-balloons that are compounded into the adhesive agent layer 8 have compressibility properties and then after removing the pressure, they demonstrate regeneration properties, and the foam shape is assumed, and because of that this tape demonstrates excellent adhesive properties relative to concrete surface etc., surfaces containing protrusions and indentations. Also, in the case of the polyurethane powder 5, at the time of pull-out processing by using a BIC blade etc., the adhesion of the adhesive agent to the blade, is eliminated. Especially, because of the fact that the surface of the adhesive agent layer 6, passed the reinforced substrate material 3, is smooth, the reinforced substrate material 3 and the adhesive agent layer 6, become the skin layer.

[0016]

Regarding the thickness of the adhesive tape 2, it is affected by the particle diameter of the micro-balloons and the polyolefin powder 5, the thickness of the substrate material, etc., however, if it is at or above 150 microns, and preferably at or above 250 microns, it is possible to obtain an adhesive tape which has especially high compressibility properties, regeneration properties, and adhesive strength.

[0017]

The thickness of the adhesive agent layer 6, that is formed passed the substrate material depends on the size of the pores of the used substrate material, however, it is most appropriate if it is in the range of 10 ~ 100 microns. If it is less than 10 microns, the adhesive properties are poor, and if it is at or above 100 microns, at the time of the pull-out processing of the adhesive tape, it is easy for the adhesive agent to adhere on the BIC blade.

[0018]

Regarding the manufacturing method for the preparation of the adhesive tape according to the present invention, for example, as it is shown according to the presented in Figure 2, the ultraviolet light curable type adhesive agent composition material 2X is coated on the release substrate material 1, and on the surface of the above adhesive agent composition material 2X, the reinforced substrate material 3 and the transparent release film 9, are sequentially layer laminated, and this is then passed through the gap between rolls that are separated at a constant thickness, and the thickness of the adhesive agent layer 2 is made to be constant, and after that, ultraviolet light is irradiated and after that, the above adhesive agent composition material 2X is cured.

[0019]

At the time when the thickness of the adhesive agent layer is made to be constant through the pressure, the existing in the ultraviolet light curable type adhesive agent composition material micro-balloons 4, which are formed from elastic rubber and/or resin, and the

polyolefin powder 5, do not pass through the reinforced substrate material 3. The front surface of the adhesive agent layer 6 that has passed through the reinforced substrate material 3 is formed as a smooth surface in order to come in contact with the release substrate material and the release film material.

[0020]

Regarding the release substrate material, a material is used that is obtained as both surfaces of polyester film, etc., plastic film or paper, etc., are treated by using a silicone type release treatment agent. As the film or paper, that becomes the base, it is possible to use transparent or colorless, material, that has a brightness of 6 or less, or a colored material where the colored color rings is from blue to green color. Here, regarding the colorless material with brightness of 6 or less, it is the range of colors from light ash color (brightness of 6) to black color (brightness of 1). The dark ash color has a brightness in the range of 3 ~ 5. Also, as the color of the colored rings that is from blue to green, for example, it is preferred to be the 5PB ~ 5BG blue color according to the Mancel color ring. If colored plastic films or paper besides the above described are used, there is the trend that the thermal resistance maintaining strength of the surface of the above adhesive agent layer that is in contact with this surface becomes significantly decreased.

[0021]

On the other hand, the reinforced substrate material 3 is impregnated with the ultraviolet curable type resin composition solution A, however it is preferred that it is a material where the micro-balloons 4, which are formed from elastic rubber and/or resin, and the polyolefin powder 5, have not passed through. Because of that, the particle diameter of the micro-balloons 4 and the polyolefin powder 5, of the used materials is larger than the porosity of the synthetic fiber, natural fiber or glass fiber, etc., woven materials, non woven materials, Japanese paper, etc., that are used as the reinforced substrate material 3.

[0022]

Among these, the nonwoven fabric material where jute and Rayon have been mixed, is the most preferred, and it is appropriately selected by taking into consideration the particle diameter of the micro-balloons 4 and the polyolefin powder 5, and the viscosity of the ultraviolet light curable type adhesive agent.

[0023]

Regarding the micro-balloons 4, that are formed from elastic rubber and/or resin material, and that are used according to the present invention, they can be manufactured according to the manufacturing methods disclosed in the US Patents 3,615,972, US Patent 4,075,138, US Patent 4,287,308, etc.

[0024]

It is also possible to use micro-capsules where vinylidene chloride – acrylonitrile copolymer resin, or methacrylonitrile – acrylonitrile copolymer resin, etc., are used as the wall layer material, and they enclose a low boiling point hydrocarbon material. Also, it is possible to use micro-balloons made from urethane rubber as the wall layer is made from an O/W emulsion using urethane prepolymer material.

[0025]

Regarding the micro-balloons, they are micro-balloons whose wall layer is solvent resistant, so that they can be dispersed into the ultraviolet light curable type adhesive agent, and they are thermally resistant in order to improve the thermal resistance properties of the adhesive agent layer 2, and for example, the Expancel 091 DE, manufactured by Nobel Industries Company (Sweden), which is a methacrylonitrile – acrylonitrile copolymer resin, is preferred.

[0026]

It is necessary that the particle diameter of the micro-balloons is larger than the pores of the reinforced substrate material 3. If these are too large, it is not possible that the adhesive agent layer 2 is made to be a thin layer. As the particle diameter of the micro-balloons, it is preferred to have micro-balloons with particle diameters that are in the range of 20 ~ 100 microns. If the compressibility properties and the regeneration properties of the adhesive material layer 2, are considered, it is especially preferred that the particle diameter of the micro-balloons be within the range of 60 ~ 80 microns.

[0027]

Regarding the amount used of the micro-balloons formed from elastic rubber and/or resin, according to the present invention, in order that it be compressible under the pressure of 500 grf/cm or less, which is the optimum pressure during the adhesion operation, and also, in order that it is easily regenerated after the removal of the pressure, it is preferred that the amount contained in the adhesive agent layer be within the range of 10 ~ 50 volume %, and it is even more preferable, that it be within the range of 20 ~ 40 volume %. If it is less than 10 volume %, it is not possible to be sufficiently compressed, and also, if it exceeds 50 volume %, the mechanical strength of the adhesive agent layer 8, is decreased.

[0028]

Regarding the polyolefin powder, for example, polyethylene or polypropylene fine particles, prevent the adhesion of the adhesive agent to the blade at the time of the pulling technological process of the adhesive tape. If the thickness of the adhesive agent layer is also considered, it is preferred that the average particle diameter be within the range of 20 ~ 100 microns. Regarding the polyolefin powder, it is preferred that it is a material where the transmission of ultraviolet light with a wavelength of 300 nm or higher, is easy, and that it is an material that has excellent thermal resistance properties. The Miperon XM-

220, manufactured by Mitsui Petro-Chemical Company, ultra-high molecular weight polyethylene powder, which has an average particle diameter of 30 microns, etc., are preferred. Regarding the used amount of the polyolefin powder, it is preferred that it be within the range of 5 ~ 20 volume % within the adhesive agent layer. If the amount used is less than 5 volume %, it becomes easy for the adhesive agent to adhere to the blade at the time of the pulling processing, and if 20 volume % are exceeded, the adhesive agent layer becomes hard and the adhesive strength is decreased.

[0029]

Regarding the ultraviolet light curable type adhesive agent according to the present invention, it is a material that is obtained as to the main composition material of the adhesive agent, which is obtained from an acrylic acid ester of alkyl alcohol, where the number of the carbon atoms is in the range of 4 ~ 14, a compound that is copolymerizable with the above described acrylic acid ester, and a polyfunctional oligo-acrylate with a molecular weight of at least 300 or higher, and then a photopolymerization initiation agent, micro-balloons with a particle diameter in the range of 20 ~ 100 microns, and polyolefin powder with an average particle diameter in the range of 20 ~ 100 microns, are admixed.

[0030]

Regarding the acrylic acid ester of alkyl alcohol with a number of carbon atoms in the range of 4 ~ 14, it is an ester of alkyl alcohol where the number of the carbon atoms is in the range of 4 ~ 14, and acrylic acid, and for example, it is a material that can be obtained by using butyl acrylate, isoamyl acrylate, 2-ethyl hexyl acrylate, nonyl acrylate, iso-nonyl acrylate, iso-octyl acrylate, lauryl acrylate, decyl acrylate, etc.

[0031]

As the compound that is copolymerizable with such acrylic acid ester, for example, it is possible to use acrylic acid, cyclic alkyl or acrylic acid ester with a number of carbon atoms of 3 or less, and other than that acrylic acid derivative compounds, and in more details, it is possible to use ethyl acrylate, 2-hydroxy ethyl acrylate, 2-hydroxy propyl acrylate, tetra furfuryl acrylate, iso-bonyl acrylate, ethoxy ethyl acrylate, butoxy ethyl acrylate, phenoxy ethyl acrylate, phenyl acrylate, cyclo hexyl acrylate, etc. Also, it is possible to use nonyl phenoxy polyethylene glycol acrylate, or nonyl phenoxy polypropylene glycol acrylate, etc., oligo ester acrylates, etc.

[0032]

Also, if these monomers and the monomer of the acrylic acid ester of alkyl alcohol with a number of carbon atoms in the range of 4 ~ 14, are copolymerized, the glass transition point of the obtained copolymer material is increased, and because of that, the used amount of these monomers, is preferred to be adjusted so that the glass transition point of the copolymer material does not become -15°C or higher. If the glass transition point of

the copolymer material becomes -150°C or higher, the tack at normal temperature is decreased and because of that it is not preferred.

[0033]

The polyfunctional oligo-acrylate with a molecular weight of 300 or higher, which is used according to the present invention, is an ester of 2 or more acrylic acids and a polyol. This polyfunctional oligo-acrylate is preferred to be a material where the number of the carbon atoms that are between the two ester radicals and the vinyl radical, is at least 10 or higher. Such polyfunctional oligo-acrylate is used in order to increase the cohesive strength of the adhesive agent at high temperatures. In the case of the materials that are polyfunctional oligo-acrylates but have a molecular weight of 300 or less, the distance between the crosslinking molecules becomes too short and because of that when a small amount is used a large effect is imparted onto the cohesive strength, and it is difficult to control the cohesive strength and that is why it is not a preferred option.

[0034]

As the polyfunctional oligo-acrylates with a molecular weight of 300 or higher, for example, it is possible to use the following materials: ennea ethylene glycol diacrylate (NK ester A-400, manufactured by Shin Nakamura Chemical Company), tetra - deca ethylene glycol diacrylate (NK ester A-600, manufactured by Shin Nakamura Chemical Company), etc., polyalkylene glycol diacrylates, ethylene oxide modified bis Phenol A diacrylate (BP-4EA, manufactured by Kyoei Yuno Chemical Company), propylene oxide modified bis phenol A diacrylate (BP-4PA, manufactured by Kyoei Yuno Chemical Company), ethylene oxide modified trimethylol propane triacrylate (TPM-6EO-3A, manufactured by Kyoei Yuno Chemical Company), hydroxy pibaric acid neopentyl glycol diacrylate (KAYARAD MANDA, manufactured by Nippon Pharmaceutical Industries Company) and their derivatives (KAYARAD HX-620, manufactured by Nippon Pharmaceutical Industries Company), etc.

[0035]

Regarding the compounded amount of the polyfunctional oligo-acrylate with a molecular weight of 300 or higher, into the ultraviolet light curable type adhesive agent composition material, relative to 100 weight parts of the total amount of the acrylic acid ester with an alkyl alcohol with a number of carbon atoms in the range of 4 ~ 14, and the compound that is copolymerizable with that acrylic acid ester, is preferred to be within the range of 0.0001 ~ 0.002 moles. If the amount that is compounded is less than 0.0001 moles, the thermal resistance maintaining strength of the adhesive tape is decreased, and if the amount added is more than 0.002 moles, there is the trend that the adhesive tape tack would be decreased.

[0036]

As the photopolymerization initiation agent, in order to make the polymerization rate fast and also to make the polymerization degree be close to 100 %, aceto phenone type and benzo phenone type materials, are preferred, and as such photopolymerization initiation agents, it is possible to use the commercially available materials. For example, it is possible to use 4-phenoxy dicyclo aceto phenone 4- t – butyl – dichloro acetophenone, diethoxy aceto phenone, 2-hydroxy 2 – methyl – 1 – phenyl propane – 1- on, 1-hydroxy cyclo hexyl phenyl ketone, benzoin, benzoyl methyl ether, benzoin iso butyl ether, benzyl dimethyl ketal, etc., and their mixed materials.

[0037]

The amount of the photopolymerization initiation agent added into the ultraviolet light curable type adhesive agent composition material, relative to 100 weight parts of the total amount of the acrylic acid ester with an alkyl alcohol with a number of carbon atoms in the range of 4 ~ 14, the compound that is copolymerizable with that acrylic acid ester, and the polyfunctional oligo-acrylate whose molecular weight is 300 higher, is preferred to be within the range of 0.005 ~ 0.01 weight parts.

[0038]

On the other hand, the viscosity of the solution of the ultraviolet light curable type adhesive agent composition material is low, in the range of 2 ~ 20 cps, and in order to improve the coatability properties, one part of the above described ultraviolet light curable type adhesive agent composition material is subjected in advance to pre-polymerization, or a polymer material that is compatible with the monomer component is dissolved and the viscosity of the composition material is increased to be within the range of 1000 ~ 3000 cps. As the polymer materials with compatibility properties, for example, it is possible to use acrylic rubber, styrene – butadiene rubber, isobutylene rubber, styrene isoprene block polymer, styrene butadiene block polymer, low molecular weight polyethylene, low molecular weight polypropylene, etc. Also, regarding the amount of this polymer used, it is preferred to be in the range of 1 ~ 15 weight % in the used ultraviolet light curable type adhesive agent composition material.

[0039]

In the ultraviolet light curable type adhesive agent composition material used according to the present invention, it is also a good option if a small amount of pigment or dye material is added in order to color the material, as long as that does not impede the ultraviolet light transmission.

[0040]

Manufacturing of the adhesive tape

On one side of a release type substrate material, obtained as a transparent, 50 micron thick and 300 mm wide PET film has been subjected to a treatment using a silicone type

release agent, the ultraviolet light curable type adhesive agent composition material 2X is coated at a thickness in the range of 0.01 ~ 3.0 mm, and on the surface of that the reinforced substrate material 3 and the release treated surface of the 50 micron thick, 300 mm wide transparent PET film, that has been treated on one surface by using a silicone release agent, are sequentially layer laminated. The above laminated layer material is passed through the gap between two rolls, which is adjusted to be at a pre-determined thickness, and the ultraviolet light curable type adhesive agent composition material 2X is formed at the pre-determined thickness.

[0041]

First, the above described laminated layer material is passed inside an irradiation oven where 6 fluorescent light lamps with bug shields (main wavelength 352 nm, 0.44 mW/cm²), are positioned in the width direction at a distance of 60 mm, in the direction that is in parallel to the flow of the coated layer, and irradiation is conducted from a height of 200 mm relative to the material and an ultraviolet light irradiation at approximately 2.00 mW/cm², was conducted. By using this irradiation equipment, the ultraviolet light irradiation relative to the laminated material formed from the shown in Figure 2, release substrate material 1, ultraviolet light curable type adhesive agent composition material 2X, reinforced substrate material 3, release transparent film 9, is conducted from the side of the release, transparent film 9. It is preferred that through this irradiation at least 80 % or more of the monomer inside the ultraviolet light curable type adhesive agent composition material 2X, is polymerized.

[0042]

After that, by using a second irradiation oven, which utilizes a high pressure mercury lamp with a main wavelength of 356 nm (for example, at least 30 mW/cm² or higher level), an ultraviolet light irradiation is conducted, and the polymerization is completed. In this second irradiation oven, it is preferred that a ozoneless type lamp be used, which does not generate ultraviolet light in the low wavelength region of 200 nm or less. The low wavelength region ultraviolet light are harmful because of the fact that they ozonate the oxygen in the atmosphere, and also, they deteriorate the adhesive tape and that is why they are not preferred.

[0043]

In the case when the cure is conducted by using only one radiation oven, there is the trend that the tack of the obtained adhesive tape is decreased, however, by curing using a first radiation oven and a second radiation oven, by the irradiation of the material as it is tacky, the increase of the tacky properties becomes possible.

[0044]

[Effect]

In the case of the adhesive tape according to the present invention, through the micro-balloons 4, which are formed from elastic rubber and/or resin, and that are present inside the adhesive agent layer 8, which is formed on the surface of the release type substrate material, the adhesive agent layer 2 is a material that can be compressed and that can regenerate approximately the same thickness as the original thickness after the removal of the pressure. Also, the adhesive agent layer 8, is a foam type material, and there is an adhesive agent which flows even into the indented parts of the material subject to the adhesion, which has protrusions and indentations, relative to the adhesive agent layer 6, that has passed through the reinforced substrate material 3, and because of that it is possible to obtain a strong adhesion.

[0045]

Regarding the polyolefin powder 5, which is present in the adhesive agent layer 8, it is a material, which can increase the manufacturing properties as the processing properties are improved because of the fact that the adhesion of the adhesive agent onto the blade at the time of the adhesive tape pulling processing, is suppressed. Also, if the solventless ultraviolet light curable type adhesive agent composition material according to the present invention is obtained, there is no pollution of the environment, and it is possible to manufacture the adhesive tape according to a method that is inexpensive and that has little fire risk.

[0046]

[Practical Examples]

Here below, the present invention will be explained based on practical examples.

Practical Example 1

To 100 grams of a mixed monomer solution comprised of 90 grams of 2-ethyl hexyl acrylate, and 10 grams of acrylic acid, 3 grams of acrylic rubber (Toakuron PS220, manufactured by Toa Paint Company) are mixed and dissolved by stirring for a period of 48 hours in a vessel equipped with a stirring device. This solution had a viscosity of 750 CPS.

[0047]

To this solution, as a polyfunctional oligo-acrylate, 0.0005 moles (0.156 grams) of hydroxy pibaric acid neopentyl diacrylate with a molecular weight of 312 (KAYRAD MANDA, manufactured by Nippon Pharmaceutical Industries company) and 0.05 grams of 2-hydroxy 2-methyl -1-phenyl propane 1-on (Darocure 1173, manufactured by Merck Company), used as an acetophenone type photopolymerization initiation agent, were compounded.

[0048]

Especially, 1.0 grams of micro-balloons, which have as their wall layer, a copolymer material obtained from methacrylonitrile and acrylonitrile, and have an average particle diameter in the range of 60 ~ 80 microns, and a true density of 0.025 g/cm³ (Expancel 1091, manufactured by Nobel Industries Company), and 10 grams of polyolefin powder, with an average particle diameter of 30 microns, and a density of 0.94 g/cm³ (Miperon XM-220, manufactured by Mitsui Chemical Company), were added. According to the calculations based on the density, the volume taken up by each the Expancel and the Miperon XM-200, inside the adhesive agent is correspondingly, approximately 35 volume %, and 9 volume %. And these were well dispersed and after that, the material was left to stay and it was de-foamed, and the ultraviolet light curable type adhesive agent composition material, was obtained.

[0049]

After that, by using the obtained ultraviolet light curable type adhesive agent composition material the described here below adhesive tape was manufactured. First, on one side of a double side release substrate material, obtained as both sides of a transparent polyester (PET) film with a thickness of 50 microns have been treated by using silicone type release agent, the ultraviolet light curable type adhesive agent composition material was coated so that the thickness becomes approximately 0.7 mm. After that, on its surface the reinforced substrate material (nonwoven fabric material with a thickness of approximately 20 microns, Napkin B2, manufactured by Daifuku Paper Manufacturing Company) and transparent release film obtained as one side of the 50 micron thick transparent PET film has been treated by using a silicone type release agent, were sequentially layer laminated and after that, this material was passed through the gap between two rolls that have been adjusted to a pre-determined gap, and the adhesive agent composition material was made to be a layer with a thickness of 0.7 mm. By this compression, the adhesive agent is impregnated into the nonwoven fabric material, and its passing through the reinforced substrate material is observed.

[0050]

After that, the above described laminated layer material is passed inside an irradiation oven where 6 fluorescent light lamps with bug shields (main wavelength 352 nm, 0.44 mW/cm²), are positioned in the width direction at a distance of 60 mm, in the direction that is in parallel to the flow of the coated layer, and irradiation is conducted from a height of 200 mm relative to the material and an ultraviolet light irradiation at approximately 2.00 mW/cm², was conducted from the side of the transparent PET film side for a period of 60 seconds. Especially, by using a high pressure mercury lamp (with a main wavelength of 356 nm and an output of 80 W/cm), an ultraviolet light irradiation is conducted for a period of 30 seconds at a irradiation intensity of 30 seconds, and an adhesive tape was obtained where the ultraviolet light curable type adhesive agent composition material was sufficiently cured.

[0051]

The thickness of the adhesive tape was approximately 0.710 mm, and when the cross sectional surface of the adhesive tape was observed by using a high magnification microscope, the in the space between the reinforced substrate material and the transparent release film, Expancel 1091 DE and Miperon XM-220, were not observed, and the front surface of the adhesive agent layer, was smooth. The nonwoven fabric interface was not separated, however, the thickness of the adhesive agent layer that has been exuded from the substrate material was approximately 20 microns.

[0052]

The evaluation of the obtained adhesive tape was conducted according to the following.

(1) Polymerization ratio

From a sample formed from two types of adhesive agent layers, that was subjected to ultraviolet light irradiation by using fluorescent lamps with bug shields for a period of 60 seconds and that, especially, was subjected to an ultraviolet light irradiation by using high pressure mercury lamp for a period of 30 seconds, approximately 0.50 grams were weighed, and this was left to stand at a temperature of 120°C and a pressure of 5 mm Hg for a period of 2 hours, and the volatile components were volatilized and the reduction of the weight was monitored. This weight reduction amount was taken as the non-polymerized monomer material, and the polymerization ratio was calculated according to the described here below formula.

Polymerization ratio = $[1 - (\text{weight reduction amount} / \text{weight of the adhesive agent prior to the treatment})] \times 100$

According to this result, the polymerization ratio of the adhesive agent after the irradiation by the fluorescent lamps equipped with bug shields, was 79.5 %, and especially, the polymerization ratio after the irradiation using the high pressure mercury lamp was 99.6 %.

[0053]

(2) Compression, regeneration testing

The adhesive tape is glued onto a 5 mm thick flat plate, and on the adhesive agent layer where the release type substrate material has been removed, a cylindrical part 500 gram weight metal block, with a cross sectional surface of 1 cm², was placed, and at a temperature of 23°C the material was compressed for a period of about 30 seconds, and then the sinking depth (thickness) of the adhesive agent layer, was measured, and immediately the load was removed and the thickness of the regenerated adhesive tape was measured after 1 hour. These results show that the thickness after 1 hour was 0.695 mm, and the distance of 0.3 mm, which was due to the sinking by the compression, was regenerated to 97.9 %.

(3) Elongation ratio

The adhesive tape was cut to a width of 8 mm and a length of approximately 100 mm, and the elongation ratio was measured at an extension rate of 20 mm/minute. The results showed that the elongation ratio was 25 %.

(4) Pulling processing testing

An adhesive tape that is inserted between a release treated craft paper, where one side has been separated, and a separated PET film, was pulled and processed by a BIC die. When it was continuously pulled and processed through 100 shot circle with a diameter of 20 mm, there was no adhesion of the adhesive agent on the blade of the BIC die.

(5) Measurement of the thermal resistance maintaining strength

The measurement of the holding strength was conducted according to the holding strength measurement method in the JIS Z 0237. In this case, as the material subject to the adhesion, a stainless steel plate with a thickness of 2mm, that has been polished by using number 280 water resistant polishing paper, and a 0.05 mm thick aluminum foil, were used. On the aluminum foil a double sided adhesive tape was glued so that the surface area became 25 mm x 50 mm or higher, and then the adhesive agent layer was placed in between so that the surface area of contact with the steel plate became 25 mm x 25 mm, and an aluminum foil was overlapped, and adhered. In order to observe the adhesive strength relative to the stainless steel plate, the adhesive surface area relative to the stainless steel was made to be small. The measurement was conducted on both sides of the adhesive tape. As a result from that it was found that in the case of both distances, it was 0.2 mm.

(6) Adhesive strength relative to rough surface containing protrusions and indentations (undulated surface)

A wall material was used as the material subject to the adhesion. This wall material is a siding board that has been blow painted by using an acrylic paint material, and the size of the protrusions were a diameter of 1 ~ 7 mm and a height of 0.5 ~ 1 mm. This wall material was cut into dimensions of width of 30 mm and a length of 200 mm. The adhesive tape was cut to a width of 20 mm and a length of 100 mm, and the foam type adhesive agent layer surface was lightly glued onto the wall material. Also, an aluminum foil with a thickness of 50 microns, that has been cut to a width of 30 mm and a length of 200 mm, was used as the backing material, and on the surface of that a roller with a weight of approximately 2 kg, was passed back and forth one time and by that it was adhered. After the adhesion, the material was left to stand at a temperature of 23°C for a period of 48 hours, and the peel strength was measured as it was peeled at a peeling rate of 300 mm/minutes, in a direction at an angle of 90 degrees. As a result from that it was found that the adhesive agent was strongly adhered onto the wall material, and the adhesive agent underwent a cohesive failure over the whole surface. This is because of the fact that the adhesive agent layer that is compressed by the pressure adhesion

operation is intimately adhered onto the surface of the wall which contains protrusions and indentations. The adhesive strength was 4.0 kgf/cm.

[0054]

Practical Example 2

In a mixed monomer material obtained from 9 kg of 2-ethyl hexyl acrylate, and 1 kg of acrylic acid, 1.5 grams of photopolymerization agent 2-hydroxy 2-methyl-1- phenyl propane 1-on (Darocure 1173, manufactured by Merck Company), were dissolved. Especially, 100 grams of the Expancel 1091 DE and 1 kg of the Miperon XM-220, used according to the Practical Example 1, were dispersed. The calculations based on the density indicated correspondingly that the Expancel 1091 DE takes up approximately 35 volume %, and the Miperon XM-220, takes up approximately 9 volume %.

[0055]

This monomer dispersion material was placed into an ultraviolet light radiation reaction vessel with a shutter structure, and by using nitrogen gas, the dissolved oxygen and the oxygen in the ambient atmosphere was chased out, and then irradiation by using ultraviolet irradiation was conducted until the viscosity was increased to 700 CPS. After that, as a polyfunctional oligo-acrylate, 20 grams of hydroxy pibaric acid neopentyl diacrylate with a molecular weight of 312 (KAYRAD MANDA) and, especially, 15 grams of 2-hydroxy 2-methyl -1-phenyl propane 1-on (Darocure 1173), used as photopolymerization initiation agent, were added and the ultraviolet light curable type adhesive agent composition was manufactured.

[0056]

After that, the same procedures as those described according to the Practical Example 1, were followed and an adhesive agent layer was formed on the surface of a PET film, that has been release treated on both sides, and an ultraviolet light irradiation was conducted and an adhesive tape with a thickness of 0.705 mm, was manufactured. The front surface of the adhesive tape was smooth, and only adhesive agent has passed through the reinforced substrate material, and its thickness was approximately 25 microns. Also, as a result from the evaluation, which was conducted the same way as described in the Practical Example 1, it was found that the polymerization ratio was 99.8 %, that the sinking under a load of 500 ggr/cm², was 0.28 mm, and that the regeneration coefficient was 98.0 %, and the elongation ratio was 35 %. Especially, the pulling processing properties also were good and there was no adhesion of the adhesive agent onto the BIC blade observed. Especially, the thermal resistant holding strength of the irradiated surface and of the opposite surface were each below 0.1 mm and there was no difference observed, and the adhesive strength relative to a surface containing protrusions and indentations was 4.1 kgf/cm, and there was a cohesive failure.

[0057]

Reference Example 1

Solvent type adhesive agent

To 1000 grams of a monomer mixture formed from 93 mole % of 2-ethyl hexyl acrylate, 5 mole % of acrylic acid, 2 mole % of 2-hydroxy ethyl acrylate, 3 grams of benzoyl peroxide, were added. In a 3 liter separable flask with three openings, equipped with a cooling condenser, and a stirring device, 400 grams of the monomer mixture with the dissolved in it benzoyl peroxide and 600 grams of ethyl acetate, were introduced, and the atmosphere was sufficiently exchanged with nitrogen, and it was recirculated as the temperature was gradually increased. The recirculation temperature was 76°C. The recirculation continued for a period of 1 hour, and the remaining monomer mixture was added in parts within a period of 2 hours. After that, the recirculation continued for a period of 3 hours, and especially, 2730 grams of ethyl acetate, were added, and a viscous acrylic polymer solution, with a viscosity of 3200 CPS, was obtained. After that, to 100 grams of this acrylic polymer solution, 100 grams of xylene resin (H-80, manufactured by Nippon Carbide Company), and 6 grams of curing agent (Coronate L, manufactured by Nippon Polyurethane Company), were added, and this was sufficiently mixed and an adhesive agent solution, was obtained. The solid phase of this adhesive agent solution was 29.5 %. So that at a volume % relative to the solid parts of 100 g of this adhesive agent solution, that is the same as that in the case of the Practical Example 1, 0.26 grams of micro-balloons, which have as their wall layer, a copolymer material obtained from methacrylonitrile and acrylonitrile, and have an average particle diameter in the range of 60 ~ 80 microns, and a true density of 0.025 g/cm³, Expancel 1091, and 2.65 grams of polyolefin powder Miperon XM-220 (with an average particle diameter of 30 microns, and a density of 0.94 g/cm³), were added and dispersed well. This was de-foamed as it was left to stand, and a viscous solvent agent type adhesive agent solution, was obtained, which contained compounded hollow micro-balloons and polyolefin powder, and which had a viscosity of 4000 CPS.

[0058]

After that, the double-sided release treated PET film and the substrate material, used according to the Practical Example 1, were used and an adhesive tape was obtained according to the described here below method. On the surface of the PET film the substrate material was placed, and then on the surface of that the adhesive agent solution with a viscosity of 4000 CPS, was coated so that it became as thick as possible. Especially, on the adhesive agent solution, another substrate material was placed, and it was adjusted so that the thickness became homogeneous. In order to prevent the foaming at the time when the solvent agent was volatilized, this was left to stay at normal temperature for a period of 1 day, and after that, a treatment was conducted at a temperature of 130°C for a period of 10 minutes, and the solvent agent was dried and the adhesive tape was obtained.

[0059]

As a result from that, despite the fact that it was left to stand at normal temperature for 1 day, during the treatment at a temperature of 130°C, there was foam generation, and the adhesive tape surface became uneven. The thickness of the adhesive tape at the parts where there was no foaming and the surface was relatively smooth, was 0.35 mm. Despite the fact that the viscosity of the adhesive agent solution was high, and that it was coated as thick as possible, it was understood that it was difficult to obtain an adhesive tape with a thickness of 0.7 mm, as in the case of the Practical Example 1.

[0060]

Reference Example 2

Adhesive tape without a substrate material

By using the ultraviolet light curable type adhesive agent composition material obtained according to the Practical Example 1, and except for the fact that there was no substrate material, everything else was conducted the same way as in the case according to the Practical Example 1, and by ultraviolet light irradiation, an adhesive tape was manufactured. As a result from that, an adhesive tape where the polymerization ratio of the adhesive tape was 99.5 %, the thickness was 0.700 mm, which is the same as that in the case of the Practical Example 1, was obtained. However, the elongation ratio of the adhesive tape was 950 %, and at the time when the adhesive tape that has been cut into a band type shape with a width of 8 mm and a length of 1 mm, was glued onto the material subject to the adhesion, the adhesive tape was soft and had no skeleton, and because of that, the gluing on the target position was difficult. Also, when once it was adhered, and after that it was separated in order to make a correction, the adhesive tape was extended, and it was not possible to be used again, and it was understood that the adhesion procedure operational properties, were deteriorated.

[0061]

Reference Example 3

The hollow micro-balloons and the polyolefin powder were not used, and an ultraviolet light curable type adhesive agent composition material according to the Practical Example, with the exception of the hollow micro-balloon Expancel 1091 DE and the polyolefin powder XM-200, was obtained. This composition material was used and by using exactly the same method as that described according to the Practical Example 1, an adhesive tape, was manufactured. As a result from that, an adhesive tape with a thickness of 0.698 mm was obtained, where the thickness of the adhesive layer that has passed through the substrate material, was approximately 20 microns. Also, the polymerization ratio was 99.9 %, however, at the time of the compression testing, the sinking depth was less than 0.2 mm, and it was almost not compressed. The elongation coefficient was approximately 30 % and the gluing procedures were easy, however, the pulling

processing testing showed that by 5 shots using the BIC blade there was adhesion of the adhesive agent.

[0062]

[Results From the Present Invention]

- (1) The foamed type surface is rich in flexibility properties and especially, it adheres well relative to a rough surface containing protrusions and indentations, like concrete, etc.
- (2) Because the skin layer has a small elongation coefficient, the adhesion procedure operational properties are excellent.
- (3) At the time when it is glued onto a material subject to the adhesion, which has a surface containing protrusions and indentations (undulated surface) or has crevices, because of the fact that the adhesive agent layer is compressible, and especially, because it has regeneration properties, it intimately contacts with the surface of the material subject to the adhesion. Because of that a strong adhesion is possible.
- (4) It is possible to be used as a foam type tape.
- (5) An adhesive tape with excellent pulling processing properties, is suggested.
- (6) Because of the fact that a substrate material is inserted into the adhesive agent layer, the elongation ratio is small, and because of that, the adhesion procedure operational properties are good.
- (7) A manufacturing method is suggested where there is no pollution of the environment and it is inexpensive and there is little fire hazard.

[0063]

[Brief Explanation of the Figures]

[Figure 1]

Figure 1 is a diagram, which represents the structure of the adhesive tape.

[Figure 2]

Figure 2 is a diagram representing the manufacturing technological processes for the preparation of the adhesive tape.

[Explanation of the Symbols]

- 1.....release substrate material
- 2.....adhesive agent layer
- 2X.....ultraviolet light curable type adhesive agent composition material
- 3.....reinforced substrate material
- 4.....micro-balloons

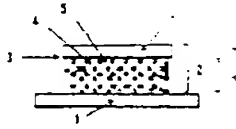
- 5.....polyolefin powder
- 6.....adhesive agent layer, that has passed through the reinforced substrate material
- 7.....skin layer
- 8.....foam type adhesive agent layer
- 9.....transparent release film

Patent Assignee: Sony Chemical Company

Translated by Albena Blagev ((651) 735-1461 (h), (651) 704-7946 (w))

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【図1】



【図2】

